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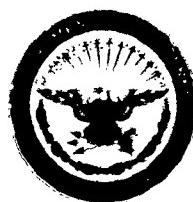
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**MATERIALS REPORT NO. 29**

**PROGRESS REPORT ON VANASIL INVESTIGATION INCLUDING  
DATA ON A LOW EXPANSION ALUMINUM POWDER ALLOY**

**PROGRESS REPORT NO. 4**

**by**

**B. C. Vaughn**



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**U. S. NAVAL ORDNANCE PLANT  
INDIANAPOLIS, INDIANA**

**MATERIALS DIVISION  
REPORT NO. 29  
PROGRESS REPORT NO. 4**

**RESEARCH AND TEST DEPARTMENT  
MATERIALS DIVISION**

**PROGRESS REPORT ON VAMAXIL INVESTIGATION INCLUDING  
DATA ON A LOW EXPANSION ALUMINUM POWDER ALLOY**

**6 May 1958**

**By: B. C. Wagner  
Metallurgical Branch**

**Approved:**

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SUBJECT: Progress Report on Vanasil Investigation Including Data on a Low Expansion Aluminum Powder Alloy

- REFERENCE:
- (a) Progress Report on Vanasil Investigation, NOPI Research Department, Report No. 16, Preliminary No. 1 by J. C. Wagner and J. G. Weir
  - (b) Progress Report No. 2 on Vanasil Investigation, NOPI Research and Test Department, Report No. 17 by J. C. Wagner and J. G. Weir
  - (c) Progress Report No. 3 on Vanasil Investigation, NOPI Research and Test Department, Report No. 21 by J. C. Wagner and B. C. Vaughn
  - (d) Some Experiments in the Production of Aluminum-Nickel-Iron Alloys by Powder Metallurgy by P. R. Kalischar, Metals Technology, August 1941, T.P. 1302
  - (e) Recent Developments in the Formation of Aluminum and Aluminum Alloys by Powder Metallurgy by G. D. Cremer and J. J. Cordiano, Metals Technology, June 1943, T.P. 1574
  - (f) Metallography of Aluminum Alloys, L. F. Mondolfo, John Wiley and Sons, 1945
  - (g) Heat Resisting Sintered Aluminum by R. Irmann, The Engineers Digest, Vol. 13 No. 1, Jan 1952, page 9

#### ABSTRACT

An aluminum powder alloy was produced with a linear coefficient of expansion of  $9.5 \times 10^{-6}$  inches per inch per degree F. ( $68^{\circ}$ - $2120^{\circ}$  F) with other physical properties equal to those of Vanasil, but with considerably improved machinability. A large number of aluminum powder alloys were surveyed in an attempt to find the most practical light alloy for use with 300 series stainless steel and phosphor bronze in instrument manufacturing.

Silicon segregation in a Vanasil casting was reduced by reducing section size of the casting and thus increasing the freezing rate of the metal.

Two trail computers built on A-132 base castings operated perfectly after exposure to temperatures of  $-60^{\circ}$  and  $165^{\circ}$  F.

#### 1. Introduction

a. The original objective of the Vanasil investigation was to develop a light weight casting alloy with the low coefficient of expansion of Vanasil but with improved machinability. The linear coefficient of thermal expansion of Vanasil is ideally suited to the manufacture of instruments in which type 303 stainless steel and phosphor bronze grade B components are assembled into an aluminum base casting alloy frame. However the difficulty of machining this material gave rise to a program aimed at developing a successor to Vanasil in such applications.

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b. Two lines of attack on this problem were suggested: first, variations of analysis of normal low expansion aluminum base alloys to reduce the primary silicon precipitation which makes Vanasil so difficult to machine; and second, development of an alloy by powder metallurgy methods to accomplish the same end.

c. A little work was started on the variation of analyses using titanium to replace the hypersubtectic portion of the silicon in Vanasil. An alloy was made with a coefficient of linear expansion of  $9.7 \times 10^{-6}$  inches per inch per degree F ( $68^{\circ}$  -  $212^{\circ}$ ). The high temperatures required to melt the titanium-aluminum master alloy made the casting very gassy in spite of the Argon atmosphere used over the whole melting and pouring equipment. Further melting of the high temperature aluminum alloys was scheduled for a vacuum furnace and until the furnace could be procured, the activity of the investigation was directed toward powder metal alloy development.

### 2. Milled Low-Expansion Aluminum Powder Alloy

a. The best powder metal alloy produced on this program was made by ball milling a powder mixture consisting of 13% silicon -325 mesh, 3.5% nickel -325 mesh, balance atomized aluminum -100 mesh. This powder was milled 20 hours under methyl alcohol in a porcelain jar with ceramic cylinders as a grinding medium. Test bars .280 x .300 inches in cross section by 3 inches long were compacted at 80,000 PSI. The compacts were sintered at temperatures of  $1000^{\circ}$ ,  $1020^{\circ}$ , and  $1040^{\circ}$  F for 1 and 2 hours in a hydrogen atmosphere dried to a dew point of  $-43^{\circ}$  F. The coefficient of thermal expansion was determined on most of the individual test bars and then their transverse breaking strength was determined by loading them as a simple beam over a 2.40 inch span. Table 1., is a survey of the properties obtained from the variations of the sintering technique.

Sample Number	Sintering			Trans. Break. Load, Lbs.	Coeff. of exp. $\text{/}^{\circ}\text{F} \times 10^{-6}$
	Time, Hrs.	Temp. $^{\circ}\text{F}$ .	Atmosphere		
V5	2	1000	H <sub>2</sub>	205	9.7
V6	2	1000	H <sub>2</sub>	203	9.8
V7	2	1000	H <sub>2</sub>	182	9.9
V8	2	1000	H <sub>2</sub>	206	9.8
X20	1	1020	H <sub>2</sub>	161*	9.6
X21	1	1020	H <sub>2</sub>	---	9.5
X22	2	1020	H <sub>2</sub>	163*	9.0
X1	1	1040	H <sub>2</sub>	198	---
X2	1	1040	H <sub>2</sub>	217	10.6
X3	2	1040	H <sub>2</sub>	226	---
X7	2	1040	H <sub>2</sub>	233	---
X8	2	1040	H <sub>2</sub>	234	9.0

TABLE 1. STRENGTH AND EXPANSION DATA ON BALL MILLED ALUMINUM POWDER ALLOY (13% Si 3.5% Ni)

\* The two 160 pound strength values of the "X-20" series are considered abnormally low and not typical of the usual production.

b. The "V" series from this table shows a high degree of consistency in strength and expansion characteristics. Both the "X" series show that increasing sintering time at a constant temperature reduces the coefficient of expansion. Over this range of temperatures and time the transverse breaking strength is not materially effected. The distortion or shrinkage of these samples increases with more severe sintering conditions but is not considered offensively high at the optimum sintering temperature of 1080° for 1 hour. Under these conditions a 3" bar will shrink .040 inches in length on sintering.

c. To compare the strength of these bars with the standard cast materials several bars were machined to the same dimensions from both as cast and heat treated Vanasil and A-132. These were then tested as beams in the same manner as the powder bars. On this basis, the powder bars of these series have about 120% of the strength of heat treated Vanasil and 91% of the strength of A-132. A number of attempts were made to test these materials as small tensile specimens. The stronger samples behaved fairly well, but a good many of the weaker samples were broken in the machine shop. Finally the transverse beam test was adopted as a standard testing method to avoid the loss of data on poorer samples.

d. The most objectionable machining operations on the Vanasil castings are drilling and tapping small holes. As a comparison, holes of equivalent size were drilled and hand tapped in some of these sinterings and in some Vanasil. The hand operations were very much easier in the sinterings than in Vanasil. No exhaustive machinability rating studies were attempted but both the actual hand operations and the microstructure of these materials show a clearly improved machining problem. This material is brittle but is certainly no worse than Vanasil in this respect.

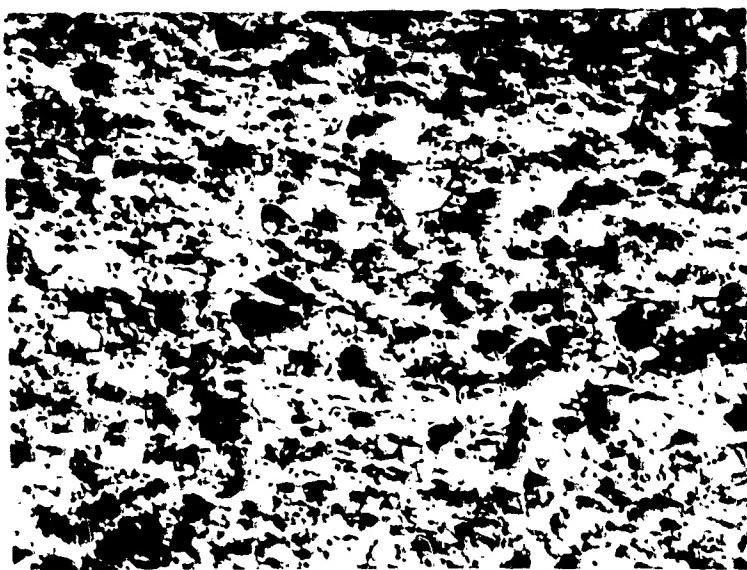


FIGURE 1. Microstructure of Ball Milled Aluminum Powder Alloy (13% Si, 3.5% Ni) X 1000. Typical of microstructures of samples listed in Table 1.

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e. The largest grain in this microstructure covers less than one five hundredth of the area of the primary silicon crystals in Vanasil (Reference (a), page 6). There is no evidence of grain growth or recrystallization in this microstructure.

f. The apparent density of this powder after milling and drying is 0.35 grams per cubic centimeter. Its compressibility is 7.6:1. Size distribution studies were started by dry screening. The -325 fraction was classified in a still water column, removing the settled fractions after measured time intervals. After drying again, these fractions were studied microscopically and an average size determined for each fraction. Now obviously there is a discontinuity at the change from screen to microscopic method; however it is felt that the data is most clearly expressed in this form. The size distribution is: + 200 mesh, 0%; + 325 mesh, 9%; 45 micron, 17%; 30 micron, 17%; 15 micron, 13%; 10 micron, 11%; 8 micron, 9%; 3 micron and finer, 24%.

g. The effect of compacting load was studied at loads both above and below 80,000 Psi. Samples of this material compacted at 67,000 Psi., showed almost identical properties to those compacted at 80,000. Earlier work with mixtures of elementary powders mixed without ball milling indicated that bars compacted at 105,000 Psi were superior to those at 80,000. The higher load was not available at this station for more detailed study. At loads of 80,000 Psi. the green density is 2.3 grams per cubic centimeter. Density after sintering increased to 2.4.

h. The physical properties of this material are comparable to those of Vanasil. The coefficient of linear expansion may be controlled between 9.0 and  $9.9 \times 10^{-6} \text{ "/"}^{\circ}\text{F}$ . ( $68^{\circ} - 212^{\circ}$ ) by appropriately controlling the sintering time and temperature as indicated in Table 1. Within the limitations of powder metallurgy parts and methods this is a solution to the original problem. Parts amenable to powder metal production may be manufactured by these techniques. This would not answer the demand for a casting material for the more involved instrument housings.

### 3. Powdered Vanasil and A-132 Modification

a. The first attempts at the powder approach to the Vanasil problem were made by ball mill powdering Vanasil and A-132 with small amounts of nickel added in the milling (Reference (c), page 11). The nickel modified Vanasil lost considerable silicon in milling and ended with an analysis of 15% silicon and 2-4% nickel. This only reduced the coefficient of expansion to  $10.9 \times 10^{-6} \text{ "/"}^{\circ}\text{F}$  which is approximately equal to that of A-132. The A-132 ball milled with nickel additions, though lower in silicon, produced a better coefficient of expansion (that is, nearer the nominal goal of  $9.5 \times 10^{-6}$ ) due to a longer sintering time and higher temperature. A brief recap of this early information is surveyed in Table 2. The relation of lower expansion characteristics to higher sintering temperatures suggested two additional short time sinters in air to check this trend. These two samples confirm this trend.

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SAMPLE TYPE	AMOUNTS	TIME, hrs.	SINTERING TEMP. °F.	STRENGTH	TRANS. BREAK.	Coeff. OF EXP.
	251 251			lb/in. <sup>2</sup>	LB.	$\times 10^{-4} \text{ "}/\text{°F}$
Powdered Vessel	15 2	3	950	H <sub>2</sub> (o)	---	10.8
" "	15 4	2	950	H <sub>2</sub> (o)	---	10.9
Powdered A-132 + Ni	9 8	3	950	H <sub>2</sub> (o)	59	9.8
" "	11 (a)	5	1025	H <sub>2</sub> (o)	51	8.2
" "	" (a)	0.5	990	AIR	71	9.4
" "	" (a)	0.5	1100	AIR	61	8.2
" "	"	---	---	---	145	9.6
Vessel, as cast (b)	"	---	---	---	151	---
" "	"	---	---	---	173	9.6
Vessel, heat treated (b)	"	---	---	---	176	---
A-132, T 551 (b)	"	---	---	---	253	11.0
					227	---

TABLE 2. COMPARISON OF STRENGTH AND THERMAL EXPANSION  
FOR CAST AND POWDER ALLOYS

- (a) Free case powder batch as the two previous samples
- (b) Cast specimens machined to same dimensions as powder bars for strength comparison.
- (c) Cylinder hydrogen as purchased.
- b. The samples sintered in cylinder hydrogen were no stronger than samples sintered a shorter time in air; in other words unpurified hydrogen is an inadequate atmosphere. From this point on the cylinder gas was purified to some extent by fixing the oxygen present in a Dectox cartridge and then drying it out in a Drierite tower to a dew point of -430 F. Table 2., also shows that higher sintering temperatures and longer sintering time produces a stronger effect on the coefficient of expansion than the silicon present in these samples does. This also suggests that the degree of oxidation of the sintering may have a profound effect on this coefficient without completely ruining the physical properties.

#### 4. Aluminum Powder Mixture Alloys

a. Another attempt was made to study the effect of sintering atmospheres, times and temperatures. The powder was mixed from elementary powders to the same chemical analysis as the Modified A-132, that is 10% Si -325 mesh, 9% Ni -325 mesh, 79% atomized Al 200 mesh (nominal). To this 2% TiH<sub>2</sub> was added to furnish nascent hydrogen for reducing aluminum oxide. The use of TiH<sub>2</sub> is established in this connection by Kalischer (Reference (d)) in sintering a magnetic powder alloy at 2190° F. His photomicrographs show an orifice network in sinterings without the hydride and show the absence of such a network when sintered with the hydride. The TiH<sub>2</sub> begins to decompose at 840° F furnishing nascent hydrogen for this reduction.

b. Three atmospheres were used in this phase, purified hydrogen, purified argon, and still air. Sintering times of 1/2, 2 and 8 hours, and sintering temperatures of 975°, 1000°, and 1025° F were studied in all three atmospheres.

c. The coefficient of expansion on this entire series was high, running from 10.8 to 11.7 x 10<sup>-6</sup> inches per inch per degree F. The samples sintered longer than a half hour produced an average strength equal to as cast Vannsil. The effects of sintering time, temperatures and atmosphere on strength are summarized in Figure 2.

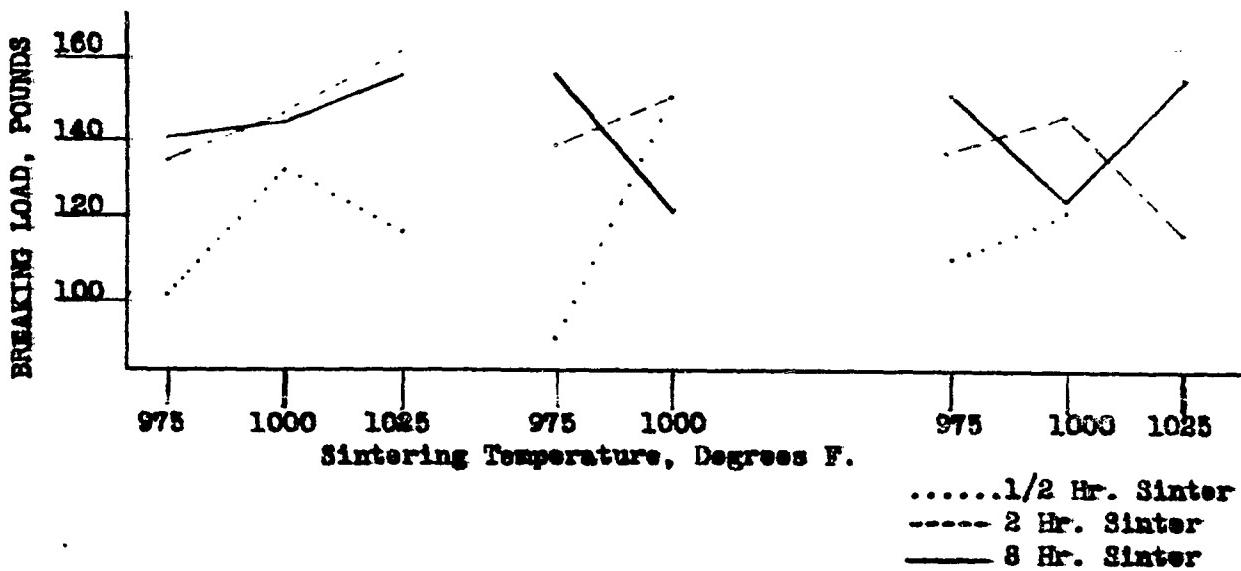


FIGURE 2. THE EFFECT OF SINTERING TIME, TEMPERATURE AND ATMOSPHERE ON STRENGTH

d. Only one good strength was attained with 30 minute sintering, so 30 minutes is considered generally inadequate. Eight hour sintering is somewhat better than two, but not enough to justify the additional trouble. Argon and hydrogen atmospheres produce comparable results under the same conditions. Neither seems very much better than air for this work.

e. Distortion of the bars due to sintering increases with higher temperatures and becomes intolerable a little above 1025°. The Al-Si-Ni system forms a eutectic at 11% Si and 5% Ni with a melting point of 1054° (Reference (f), page 114). Distortion can be expected to be severe near this temperature. In general, the highest sintering temperature consistent with reasonable dimensional stability is to be preferred.

### 5. Effect of Aluminum Powder Particle Size on Strength

a. A side study was made on the effect of powder particle size on strength alone with bars compacted from four nominal sizes of atomized aluminum powder. Nominal 20 mesh, 100 mesh, 200 mesh and -325 powders were compacted at 80,000 Psi, sintered at 1025° for 2 hours in hydrogen and tested as beams. The deflection of these beams was also measured at the moment of fracture to index the plastic deformation. This data is summarized in Table 3.

NOMINAL POWDER SIZE MESH	TRANS. BREAK. LOAD, LBS.	DEFLECTION INCHES
20	122	.420
100	148	.250
200	121	.120
-325	158	.032

TABLE 3. RELATION OF POWDER PARTICLE SIZE TO STRENGTH ON PURE ALUMINUM POWDER SINTERINGS

b. This shows an increasing strength tendency with finer powder sizes and a pronounced increase in ductility with coarser powder sizes. The best deflection on the previous alloy powder samples was .075 inches.

### 6. Effect of Titanium Hydride on Strength of Straight Aluminum Powder Bars

a. Titanium hydride, suggested by reference (d), was added to most of the alloy powder compositions in the preceding work. Some of the fractures cast a reasonable doubt as to its effectiveness in cleaning up the surface oxides on the powder grains. To check this point, a series of simple bars compacted from 20 mesh atomized aluminum powder plus from

2 to 20% TiH<sub>2</sub>, sintered 2 hours in hydrogen, was tested for comparison with the bars of straight aluminum. This data is summarized in Table 4.

AMOUNT OF TiH <sub>2</sub> ADDED %	TRANS. BREAK. LOAD, LBS.	DEFLECTION INCHES
0	122	.420
2	75	.090
4	109	.240
6	104	.180
8	66	.080
10	66	.072
20	18	.015

TABLE 4. EFFECT OF LITHIUM HYDRIDE ON STRENGTH AND EXPANSION OF PURE ALUMINUM POWDER SINTERINGS

b. No amount of added TiH<sub>2</sub> produced a strength equal to straight aluminum powder. It is concluded that sintering temperatures of 1025° F are not sufficiently high to utilize the hydrogen formed by the TiH<sub>2</sub>. Kalischer in reference (d), was sintering at 2190°F. In the light of this data, the use of TiH<sub>2</sub> was dropped from the investigation with the exception of one series of alloy samples.

#### 7. Lubrication of Dies

a. Cresser and Cordiano (reference (e)) recommended a die lubricant of flake aluminum and stearic acid or aluminum stearate in a volatile vehicle such as carbon tetrachloride to prevent the aluminum from cold welding to the die walls. Aluminum and stearic acid, half and half, suspended in acetone to make a very thin die wash worked perfectly on this investigation with a separable die.

#### 8. Complex Powder Mixture Alloys

a. One of the alloys made by replacing some of the hypereutectic silicon with titanium had a coefficient of expansion of  $9.7 \times 10^{-6}$  inches per inch per degree F. Several powder bars were mixed to this same chemical analysis to compare results with the cast alloy. Strangely, the lower sintering temperatures produced best strength, and none gave an even fair coefficient. Table 5. summarizes this information.

SAMPLE NUMBER	TIME, HRS	SINTERING TEMP. °F	ATMOSPHERE	TRANS. BREAK. LOAD, LBS.	COEFF. OF EXP. X10 <sup>-6</sup> "/"/°F
1	2	932	Dry H <sub>2</sub>	170	11.8 x 10 <sup>-6</sup>
2	2	975	" "	156	
3	2	1000	" "	125	
4	2	1000	" "	117	11.8 x 10 <sup>-6</sup>

Powder Analysis: 14% Ti; 8% Si; 2% Ni; 0.5% Cu; Balance Al

TABLE 5. STRENGTH AND EXPANSION DATA OF TITANIUM-ALUMINUM POWDER ALLOY

b. From this it appears that chemical analysis alone cannot be used to control the coefficient. The microstructure of the alloy is also important in controlling this as well as the other physical properties.

c. Next a series of bars was made to study the effect of different titanium and nickel analyses at a constant level of 8% silicon. Titanium at 5%, 14%, 20%, and 30% was mixed at nickel levels of 0%, 5%, 10% and 20% balance atomized aluminum. All were compacted at 50,000 Psi., sintered 2 hours at 1000° in dry hydrogen and tested as beams. Strength and coefficient of expansion data is summarized in Figure 3.

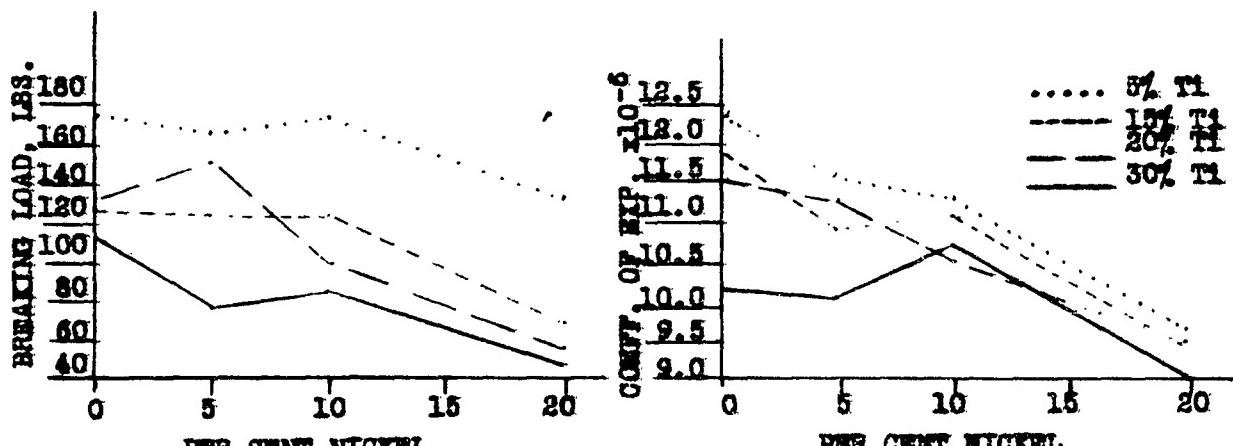


FIGURE 3. EFFECT OF TITANIUM AND NICKEL ON STRENGTH AND EXPANSION OF ALUMINUM POWDER ALLOYS

d. High percentages of nickel do reduce the coefficient of expansion but also reduce the strength to undesirable levels. The higher nickel sinterings expanded severely on sintering. So the conditions which improve the coefficient in this series ruin the strength. Of the four good coefficients produced in the series, only one equals Vanasil in strength.

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e. Several sinterings were made of 25% Si, balance atomized aluminum. These, too, were not encouraging. Strength was less than Vanasil and the coefficient more.

f. A survey of the effects of silicon, nickel, molybdenum, iron and copper on the coefficient of expansion of aluminum (reference (a)) shows that all of these elements reduce the coefficient in some degree. A comprehensive program was planned to study their effects on the coefficient and strength in powder alloys, in ternary and also in more complex composition. The general outline was to add 2% each of nickel, iron and molybdenum both singly and together to an aluminum-silicon mixture at 12%, 18%, 24% and 30% silicon levels. Each of these mixtures was checked at 1000°, 1025°, and 1050° F. sinters if it would stand it. All samples were compacted at 60,000 Psi, and sintered for 2 hours under dry hydrogen. The results on this program scattered widely, as is to be expected, and can perhaps be best summarized in Table 6.

CHEMICAL ANALYSIS					SINTERING TEMP. °F	TRANS. BREAK. LOAD, LBS.	COEFF. OF EXP. X 10 <sup>-6</sup> -"/°F.
%Si	%Ni	%Mo	%Fe	%Cu			
12	2				1000	162	11.2
12	2				1025	163	11.8
12		2			1000	158	12.1
12		2			1025	121	11.9
12		2			1050	170	11.8
12		2	2		1000	150	11.9
12		2	2		1025	145	11.9
12		2	2		1050	205	11.5
12		2	2		1000	138	11.9
12		2	2		1025	167	
12		2	2		1050	178	11.5
12	2	2	2		1000	140	11.7
12	2	2	2		1025	198	11.6
12	2	2	2		1050	78	10.8
12	2	2	2	2	1000	175	11.4
12	2	2	2	2	1025	127	11.0
18	2				1000	129	11.6
18	2				1025	132	11.4
18		2			1000	124	11.7
18		2			1025	172	11.6
18		2			1050	158	11.2
18		2	2		1000	138	11.5
18		2	2		1025	179	11.2
18		2	2		1000	150	11.5
18		2	2		1025	119	11.1
18		2	2		1050	144	11.1
18	2	2	2		1000	120	11.4
18	2	2	2		1025	130	10.9
18	2	2	2	2	1000	105	10.8
18	2	2	2	2	1025	102	10.4

(TABLE 6. continued on page 11)

(TABLE 6. continued)

CHEMICAL ANALYSIS					SINTERING TEMP. °F	TRANS. BREAK. LOAD, LBS.	COEFF. OF EXP. $\times 10^{-6}$ "/*°F
%Si	%Ni	%Mo	%Fe	%Cu			
24	2				1000	112	11.0
24	2				1025	69	11.1
24	2				1050	68	10.5
24		2			1000	97	11.4
24		2			1025	112	11.2
24		2			1050	130	11.0
24			2		1000	92	11.1
24			2		1025	120	11.0
24			2		1050	127	10.7
24		2	2		1000	113	10.9
24		2	2		1025	119	10.7
24		2	2		1050	137	10.5
24	2	2	2		1000	88	10.8
24	2	2	2		1025	98	10.6
24	2	2	2		1050	98	9.7
24	2	2	2	2	1000	88	10.5
24	2	2	2	2	1050	107	9.9
30	2				1000	47	10.8
30	2				1025	73	10.7
30	2				1050	66	9.7
30		2			1025	67	10.9
30		2			1050	119	10.1
30			2		1000	88	10.9
30			2		1025	80	10.7
30			2		1050	72	10.4
30		2	2		1000	53	10.7
30		2	2		1025	78	10.7
30		2	2		1050	108	10.4
30	2	2	2		1000	28	10.7
30	2	2	2	2	1050	56	9.2

TABLE 6. EFFECT OF ALLOY ADDITIONS TO ALUMINUM POWDER SINTERINGS

g. Only three sinterings were produced in this phase with a coefficient below  $9.9 \times 10^{-6}$  and all three of these had strengths below 100 pounds. It is fairly consistently shown that higher sintering produces higher strength. Higher sintering also improves the coefficient in practically all cases. This relation of physical properties to sintering temperature is fortunate in one respect, but up to this point sintering must be carried too dangerously near fusion to achieve adequate strength and sufficiently low a coefficient of expansion. The high temperatures required produce too much distortion to be endured before the end is reached.

## 9. Milled Aluminum Powder Mixture Alloy Development

a. In the earlier phases of this investigation a short series was run to determine the best compacting pressure for making green compacts. The analysis was the same as the other early direct powder mixture alloys, Si 10%, Ni 9%, TiH<sub>2</sub> 2%, balance atomized aluminum, however the mixing was carried out in a ball mill under alcohol for several hours. The ball milling oxidized and cold weld alloyed the powders to a greater extent than the TiH<sub>2</sub> was able to clean up. Fractures were oxidized and black.

b. Aside from the initial purpose of this experiment, it was noticed that there was a marked reduction in coefficient of expansion. This coefficient was 23% below average values for the same chemical analysis mixed without extended ball milling. Physical strength was discouragingly low, but the fact suggested an intermediate condition between the extremes of simple mixture and ball mill alloying. It appeared that the low coefficient was principally due to the oxide network which was also responsible for the low strength.

c. One possible way of improving strength at a partial sacrifice of unnecessarily low coefficient is to ball mill a master alloy under conditions that will control oxidation; then after drying the powder, blend in measured quantities of atomized aluminum to break up the continuity of oxides.

d. Several series of bars were run under such conditions. Typical of them was a series composed of 13.7% Si, 7.4% Ni, balance atomized aluminum, ball milled 20 hours under methyl alcohol in a sealed porcelain jar. After drying this powder, it was remixed, divided, and the fractions were blended with 2%, 5%, 8% and 12% 20 mesh (nominal) atomized aluminum powder. The blended powders were compacted at 80,000 Psi; half of these bars were sintered in hydrogen for 2 hours at 1000°F; the other half were sintered in hydrogen with the heat slowly increased until a liquid phase began to sweat out on the surface (at 1070°) and then were cooled in the atmosphere. The resulting coefficients and strengths are illustrated in Figure 4.

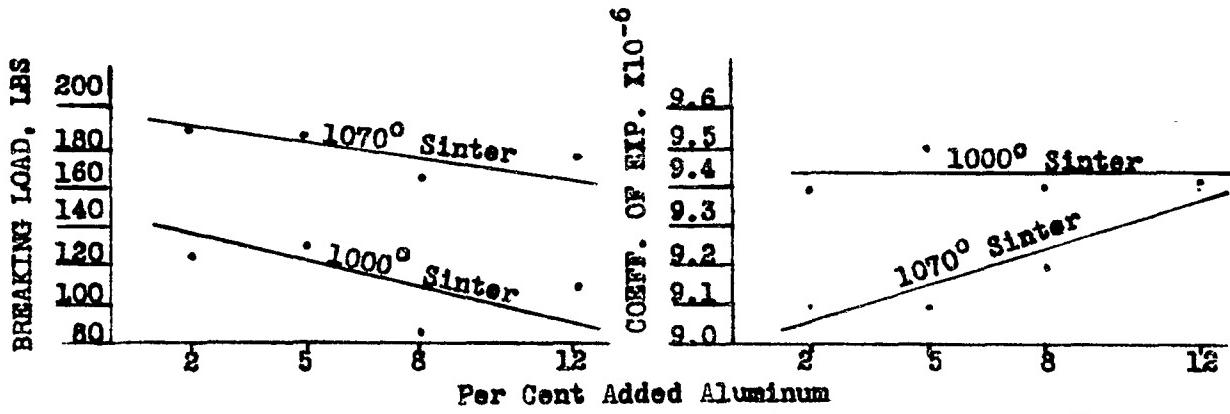


FIGURE 4. EFFECT OF ADDED ALUMINUM TO ALUMINUM-SILICON-NICKEL BALL MILLED POWDER ALLOY

e. This shows that strengths averaging slightly above Vanasil and coefficients considerably below Vanasil are possible by this method. However it is also noted that strength is reduced with added percentages of blended aluminum. This suggests that proper control of alloy composition, sintering temperature and ball milling can adequately control the relation of strength and coefficient without the addition of blended powders after milling. The data of Figure 4., also suggests that when using sintering temperatures above 1000°, there is more percentage of alloy present than necessary to reach a sufficiently low coefficient.

f. In line with these observations, the alloy was cut to 11% Si and 2.5% Ni, and the ball milling and compacting were left unchanged. Sintering was raised to 1075° and 1140° F. The blending aluminum was continued at the 2% and 8% levels in addition to the master mix without blending. This data is summarized in Figure 5.

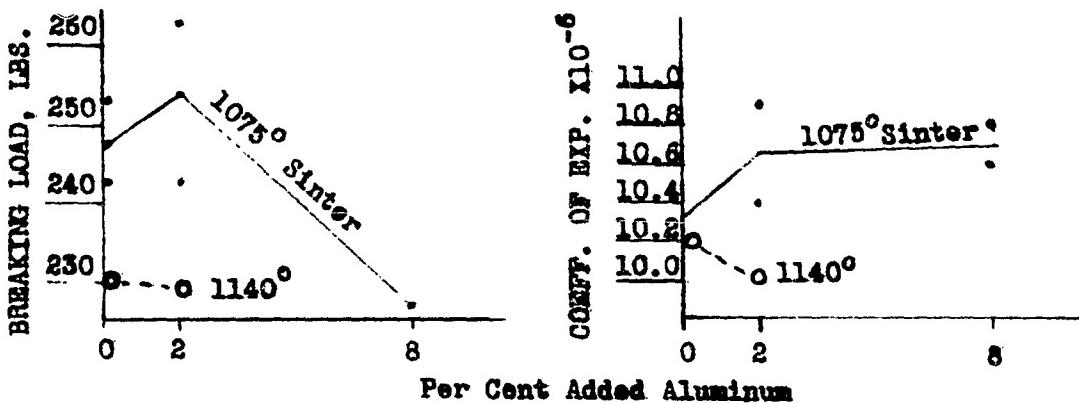


FIGURE 5. EFFECT OF ADDED ALUMINUM TO LEAN ALUMINUM-SILICON-NICKEL BALL MILLED POWDER ALLOY

g. These curves confirm the previous conclusion that little significant benefit is to be gained by blending aluminum into the master ball milled alloy. The correction on analysis however was too severe to get a good coefficient but did give the best strength yet produced (107% of A-132 or 140% of heat treated Vanasil). The higher sintering at 1140° gave a laminated, distorted bar, a very undesirable condition.

h. Two samples of this series were sintered in air at the same two times and temperatures. Both strength and coefficient were lowered, breaking load to 160 and 193 pounds and coefficient to 9.2 and  $9.9 \times 10^{-6}$  respectively at 1140° and 1075° sintering. There is an additional oxide shell or case effect which is shallow for short time sintering and at least 1/8 inch deep for 2 hour sintering. This method does not seem as promising as the hydrogen atmosphere work.

i. There is a strong possibility that hydrogen of increased purity (dew points of -60° F or more) might have improved the sintering condition for the aluminum powder mixture alloys of section 4. However it soon became apparent that high strength clean powder alloys had higher thermal expansion characteristics than alloys sintered in air or improperly purified hydrogen. This led to the conclusion that a considerable portion of the reduction in expansion was due to the presence of appreciable percentages of aluminum oxides. Of course this is particularly true in the materials prepared by extensive ball milling. Now since these properties seem to be achieved by deliberate and controlled oxidation, dew points of -43° were not found to be objectionable, particularly on the ball milled alloys.

j. This experience seems to parallel portions of the recent European developments of SAP powder products (Sintered Aluminum Powder), reference (g). The phenomenal properties of SAP products are attributed to a great extent to controlled oxidation without alloying. The coefficients of expansion of SAP material is quoted at 80% of that of EC aluminum which would be about  $10.6 \times 10^{-6} \text{ "/"/}^{\circ}\text{F}$  ( $68^{\circ}$ -  $212^{\circ}$ ). This incidentally is lower than A-132.

#### 10. Conclusions on Powder Alloy Work

a. Analysis: Several analyses were successful in reaching a linear coefficient of expansion of  $9.5 \times 10^{-6}$  inches per inch per degree F., however the most practical, simplest, and strongest found in this work was 13% silicon, 3.5% nickel, balance atomized aluminum.

b. Milling: These powders were ball milled in 180 gram sample lots in a porcelain jar, 2 1/2 gallon capacity, with 2 quarts of methyl alcohol and about 1/3 full of ceramic cylinders, for 20 hours. These conditions adequately controlled the powder grain oxidation which is largely responsible for the good coefficient. This detail would probably have to be restudied on milling in other equipment.

c. Compacting: 80,000 Psi in a separable die lubricated with a very thin wash of flake aluminum powder and stearic acid produced a nice green compact of 2.3 density. A few samples were compacted at 67,000 Psi and 53,000 Psi and a few as high as 105,000 Psi. 53,000 is too weak, 67,000 to 80,000 is adequate and 105,000, which was not available on this station, is somewhat better yet, but not essential.

d. Sintering: Since these powders are deliberately oxidized before sintering, a high purity of the atmosphere, usually required for aluminum powder metallurgy is not demanded. Hydrogen dried to dew points of -43°F is adequate. Sintering in open air further oxidized the bars near the surface, in some cases weakening them, in all cases reducing the coefficient.

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The desired coefficient may be more easily reached by this procedure, but because of the non uniform properties of center and surface, it is not recommended.

e. Dimensional Fidelity: Shrinkage on sintering is proportional to sintering severity. Increasing either time or temperature will increase the problem. However this shrinkage and its associated distortion may be held below one per cent with sinters of 1 to 2 hours and 1040° to 1080°F temperatures. This of course is not an independent variable. The coefficient is closely dependent on the sintering--longer times and higher temperatures both reduce it.

f. Machinability: The hypereutectic primary silicon crystallization is completely removed by this technique. This of course is the principal objection to Vanasil. Consequently machinability is much improved. The material is brittle, but is comparable to Vanasil in this respect-- no worse.

## II. Silicon Segregation in Slender Castings

a. One of the slender Vanasil castings used at this station consisted of a fairly plane surface a little larger than 6 x 12 inches reinforced on the back with a number of moderately heavy ribs. In the usual production of these castings the metal backing of the plane surface is .275-.285 inches thick. In this thickness there is considerable primary silicon segregation opposite the heavier ribs which would be the slowest freezing portion of the casting. This segregation is illustrated by Figure 6., an actual size photograph across the 6 inch width of the plane surface after machining.

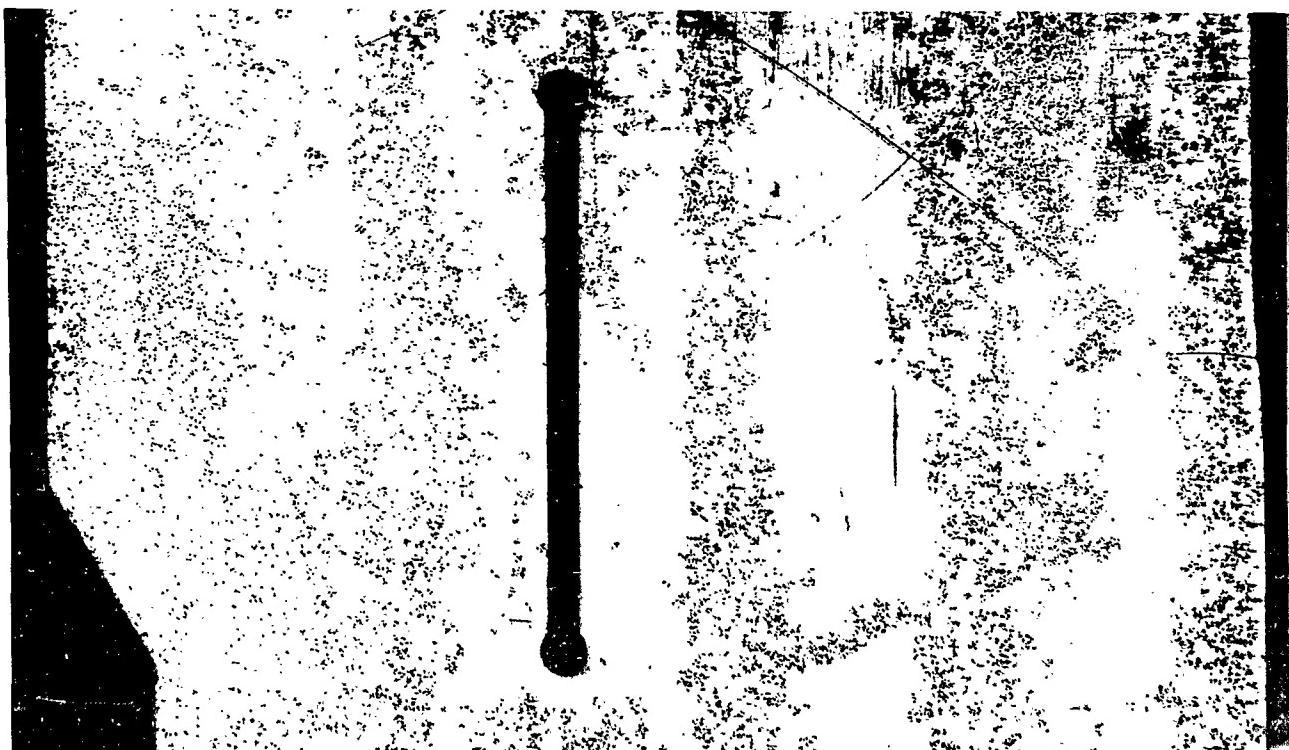


FIGURE 6. VANASIL CASTING SHOWING SILICON SEGREGATION OPPOSITE HEAVY RIBS ON BACK. DECK THICKNESS .275"-.285"

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b. The .260 inch thickness of the plane face was reduced to .220 to .230 inches thick to reduce the freezing time and thus reduce the segregation on this face. Figure 7. illustrates the success of this pattern change in reducing silicon segregation.



FIGURE 7. VANASIL CASTING SHOWING ABSENCE OF SILICON SEGREGATION AFTER REDUCING DECK THICKNESS TO .220" - .230"

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## 12. A-132 Experimental Components

a. There is considerable weight of engineering opinion favoring the use of A-132 instead of Vanasil for low expansion instrument castings. To establish the position of A-132 in the debate on this question, plans were made to build a number of the computer components of the AN/ASB-1 on A-132 frames, expose them to both high and low temperatures and then test them carefully for performance and accuracy at room temperature.

b. On this plan, two trail computers were built on A-132 base castings. They were completely tested and then exposed to temperatures from -60° to 165° F and tested again at room temperature. These computers passed all requirement both before and after the temperature cycling.

c. Continuing this plan, two time of fall computers and two V.t. units are being built for similar testing. If all satisfactorily pass these initial tests, it is planned to assemble them into a finished instrument and place them in actual service for operational observation.

d. To date no tests at this plant have shown that A-132 can be considered unsatisfactory in this application.

## 13. Recommendations for Continued Research Work to Develop Better Low Coefficient of Expansion Alloys

a. There are several avenues of possible improvement which may be suggested. It would certainly be advisable to continue the vacuum melting phase of this investigation replacing a portion of the hypereutectic silicon with titanium. The initial attempts in this direction were promising, yielding one alloy with a very favorable coefficient of expansion. It seems easily within reason that the microstructure of this alloy, or a similar one, could be controlled to a point of materially surpassing the properties of Vanasil.

b. Another suggestion that has been discussed is the possibility of refining the primary silicon crystallization of Vanasil itself by means of ultrasonic agitation of the melt either before or during freezing.

c. The similarity of SAP products and the ball milled powder alloy reported in the beginning of this report is demonstrated at least to the extent that both depend on controlled oxidation of the powder. Therefore there is a strong possibility that the techniques of hot pressing and extrusion which finish out the remarkable properties of the SAP products may also be successfully applied to this work.

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